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Rapid Gas Decompression Performance of elastomers – A study of influencing testing parameters

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Abstract

Materials used for the oil and gas industry are exposed to high pressure, high temperature and several aggressive fluids and gases. Concerning the still rising oil and gas product demand the development of new oil and gas valves is indispensable. Therefore, new reliable materials to guarantee facility safety at extreme operating conditions are needed. The presented study deals with a specific failure, the rapid gas decompression failure, which occurs due to the exposure to such extreme conditions. This failure leads to crack initiation, crack growth and in the worst case to the complete fragmentation of the component. For the characterization of this failure a hydrogenated acrylonitrile butadiene based rubber with an acrylonitrile content of 36% was exposed to several temperatures, saturation pressures, gas mixtures and different depressurization rates. Whereas, the rising testing temperature leads to decreasing volume change during the depressurization, the increase in saturation pressure, a higher decompression rate and a higher amount of carbon dioxide clearly lead to an increasing of the maximum observed volume change. Based on the observed volume change and the material ranking, determined using common testing standards NACE International (2003) and NORSOK (2001) a correlation to polymer physical principals was established.

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Keywords: rapid gas decompression, elastomer characterization, fracture of elastomers

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1. Introduction

For elastomeric materials exposed to high pressure, high temperature and different media a specific phenomenon, the rapid gas decompression failure can occur with depressurization to ambient. This specific failure leads to high volume change, crack initiation, crack growth, failure and the complete breakup of the component. Within the literature the topic was first mentioned by A.N. Gent and P.B. Lindley (1959) and B. J. Briscoe et al. (1994), and is still under discussion B. Schrittesser (2010), B. Schrittesser and G. Pinter (2011), B. Schrittesser et al. (2012), E. Ho (2006) and Z. Major and R. Lang (2009). The whole process can be divided in two separately steps, the pressurization step and the depressurization step. The pressurization step is the first step and occurs due to the penetration of different media (mainly in gas conditions) at high pressures and high temperatures. These conditions lead finally to a volume increase of e.g. up to 15%, strongly depending on temperature, pressure and the used media according to B. Schrittesser (2014). Furthermore, two effects occur during the exposure with high pressure gas. The first effect is the plasticization of the polymer matrix leading to increased backbone movement and an increasing free volume and therefore a decreasing glass transition temperature. The second effect is the compression of the polymer matrix due to the high pressure gas with the opposite behavior compared to the first effect. B. J. Briscoe et al. (1994) investigate that the nature of the gas and the applied pressure defines, if the first or the second effect is predominant. Besides the volume change and the gas sorption the matrix plasticization / matrix compression leads to a change of the mechanical and thermal properties as well and therefore to a new material. These entire changes, quest to a final equilibrium reaching constant material properties at the exposed temperature, pressure and gas mixture. The permeation process itself also strongly influences the material performance. Different permeation models are available in literature, describing the whole permeation process by C. J. Bodor (2011), D. A. Vorotnikov (2008, 2009), G. Menges et al. (2011), J. Comyn (1985), P. W. Atkins and J. de Paula (2006) and W. Henry (1802). Moreover, the permeation coefficient is also a function of the used rubber, different application temperatures, different amount of filler and different gases as described by K. Beck (2003) and R. Kreiselmaier (2002).

The second phase, the depressurization to ambient conditions is a highly complex phase with a finally high volume change of the component due to the pressure reduction to ambient conditions. This phase is basically described with three ideas. The depressurization to ambient conditions leads to a high volume increase and therefore a three-dimensional pneumatic tension state in the material (1). The nearly adiabatic depressurization process cools the ambient during the decompression resulting in a thermal properties profile across the specimen cross section (2). Hence, this leads to an established gradient of physical and mechanical properties due to the dissolved gas in the material and the temperature gradient (3). The different occurring processes during the depressurization lead finally to stress in the material and the rupture of the component. Nevertheless, due to the nearly incompressible material behavior of elastomeric materials the three dimensional tension state established during the decompression leads to void formation in the material. Additionally, voids are introduced due to badly wetted particles and enclosed air based on the production process. All these micro cavities and voids end in a crack initiation process, followed by crack growth and the failure of the material if a critical void diameter is reached, according to B. J. Briscoe et al. (1994).

Different publications deal with the determination of a critical void diameter based on the growth of one micro cavity e.g. A.N. Gent and P.B. Lindley (1959), B. J. Briscoe et al. (1994), C. Fond (2001) and J. Diani (2001). Nevertheless, based on the observations of J. Yamabe et al. (2011), Jaravel et al. (2011), O. Lopez-Pamies et al. (2011a) and T. Schwarz et al. (2008) several voids occur during the depressurization process and therefore an approach considering multiple voids is indispensable. Lopez-Pamies (2011a) announced a theory based on several pre-existing voids. Based on this theory a critical force is needed to start a crack growing process in the material. Using this theory, a theoretically solution for a critical cavitation surface $S(\sigma_m, \tau_1, \tau_2)$ is provided according to O. Lopez-Pamies et al. (2011b) by:

$$\begin{split} \mathcal{S}(\sigma_m, \tau_1, \tau_2) &= 8(3\sigma_m - \tau_1 - \tau_2)(3\sigma_m + 2\tau_1 - \tau_2)(3\sigma_m - \tau_1 + 2\tau_2) - \\ 729\mu^3\Psi^3(\tau_1, \tau_2) + 1458\mu^2\sigma_m\Psi^2(\tau_1, \tau_2) - \\ 108\mu(9\sigma_m^2 - \tau_1^2 - \tau_2^2 + \tau_1\tau_2)\Psi(\tau_1, \tau_2) - 216\mu^3 = 0 \end{split}$$
(1)

Whereas, σ_m is the mean stress, τ_1, τ_2 are the shear stresses and the shear modulus is represented by μ . Ψ represents a function $f(\tau_1, \tau_2)$ which is described in detail by O. Lopez-Pamies et al. (2011b). With the assumption of an axisymmetric loading and a supposed pure hydrostatic pressure the cavitation surface can be reduced to a cavitation point $\mathcal{P}(\sigma_m)$ according to O. Lopez-Pamies et al. (2011b).

$$\mathcal{P}(\sigma_m) = \mathcal{S}(\sigma_m, 0, 0) = \sigma_m - \frac{5\mu}{2} = 0 \tag{2}$$

The interesting point in this case is, that this theory based on multiple cavities in a material, lead to the exact same result as the theory established by A.N. Gent and P.B. Lindley (1959) which is based on the grow of one cavity in a bulk. This indicates that the interaction between cavities during the crack initiation and crack growth process plays only a secondary role. Furthermore, for industrial application of materials two standards are established, NACE (2003) and NORSOK (2001). Both testing standards define different testing temperatures, pressures, gases and decompression rates for unconstrained as well as constrained test setups. The material ranking is realized by an optical evaluation of the specimen cross section after the depressurization process based on ranking criteria. For example, table 1 summarizes the ranking criteria regarding the NORSOK testing standard. As depicted the best rating is indicated with a rating of 0. The pass/fail criterion is a material ranking of 3.

Table 1. Rating criteria according to NORSOK (2001) testing standard.

Description	Rating
No internal cracks, holes, or blisters of any size.	0
Less than 4 internal cracks, each shorter than 50% of the cross section, with a total crack length less than the cross section.	1
Less than 6 internal cracks, each shorter than 50% of the cross section, with a total crack length of less than 2,5 times the cross section.	2
Less than 9 internal cracks of which max. 2 cracks can have a length between 50% and 80% of the cross section.	3
More than 8 internal cracks, or one or more cracks longer than 80% of the cross section.	4
Crack(s) going through entire cross section or complete separation of the seal into fragments.	5

Within these testing standards the failure interpretation is based on the visual inspection of the specimen cross section of the depressurization process, only providing a pass/fail criterion without any details concerning the material performance and the kinetics of the volume change. Therefore, it is imperative to enhance the knowledge of the material performance during the exposure to high pressure gas to support an efficient material development process.

2. Experimental

Hydrogenated butadiene rubber with an acrylonitrile content of 36% and a hardness of 86 ShoreA was used for the experimental investigation of the testing parameters. For the realization of the different influencing parameter a autoclave testing system produced by SITEC (Sieber Engineering AG, Zürich, CH) was used. With the autoclave testing system several temperatures, different saturation pressures, various gases as well as various decompression rates were realized. To record the volume increase during the compression and during the decompression phase, the test system was equipped with a camera system by K. Lederer (2006). For the recording of the volume change during the experiment cylindrical specimens were used. With the observed change in height h_i and width d_i the specimen volume was calculated using the equation for cylindrical volume. To counter the barreling effect occurring during the high volume change, a linear approximation was made for the observed pictures during the experimental investigations. Table 2 summarizes the implemented experiments for the characterization of the influence of different testing parameters. Cylindrical specimens, with a diameter of 8mm and a height of 8mm, were used for all experimental investigations. Furthermore, component tests with O-Ring (internal diameter: 15,4mm; thickness: 5,33mm) were

implemented. After a preliminary study the saturation time (holding time) for all specimens was set to 20h to guarantee a full saturated state.

Testing parameter	Influence of pressure	Influence of gas	Influence of temperature	Influence of depressurization rate
Specimen geometry	Cylindrical 8x8mm	Cylindrical 8x8mm	Cylindrical 8x8mm	Cylindrical 8x8mm
	O-Ring	O-Ring		
Temperature	90°C	90°C	70, 90, 110°C	90°C
Pressure	50, 100, 150bar	100bar	150bar	150bar
Gas	CO_2	$CO_2 - CH_4$ mixtures	CO ₂	CO ₂
Decompression rate	100bar/min	100bar/min	100bar/min	20, 40, 60, 80, 100bar/min
Saturation time	~20h	~20h	~20h	~20h

Table 2. Overview of the different investigated testing parameters for decompression tests.

For the comparison of the different experiments some parameters and specific values were calculated due to the high amount of measured data. The experiment was divided in two parts: the compression phase and the decompression phase. For the compression phase the volume change during saturation phase ΔV_{comp} was calculated as difference of the initial specimen volume V_0 and the saturation volume V_{sat} . To characterize the decompression phase two parameters were used for the comparison of the different test configurations. The first parameter focuses on the volume change of the specimen, using the difference of the volume change during the saturation phase ΔV_{comp} and the maximum observed volume change during the depressurization V_{max} , to calculate the volume change during the depressurization phase ΔV_{decomp} . The second parameter was determined with the time until start of the volume change, t_{ini} , to describe the kinetic of the volume change for experiments with different depressurization rates.

3. Results and Discussion

Within the following chapter the different observed tendencies due to the several testing parameter will be discussed in detail. For an easier understanding of the tendencies the whole process is separated into two parts, the compression and the decompression phase. Additionally, also the observed specimen cross section is pictured at the different testing conditions and the ranking regarding the NORSOK testing standard (indicated by the numbers in the right upper corner).

3.1. Influence of the testing temperature

The influence of the testing temperature was assayed at three different temperatures (70°C, 90°C and 110°C) for the experimental material. Figure 1(a) summarizes the volume change during the compression phase for HNBR1 at a saturation pressure of 150bar with pure CO₂. Independently of the temperature the equilibrium state of the saturation was reached after 6000s. As depicted, the volume change during compression seems nearly independent of the applied testing temperature. Only small differences within the scattering range were observed. Based on the Brownian molecular motion according to A. Einstein (1905) the solubility decreases with rising temperature and therefore the volume change during compression phase should decrease as well. Due to the restricted resolution of the test equipment the systems seems not sensitive enough and therefore the results are in scattering range. The relative volume change during the depressurization depending on the testing temperature for HNBR1 is depicted in Fig. 1(b) for a saturation pressure of 150bar, pure CO₂ and a decompression rate of 100bar/min. The observed volume change during the decompression phase seems decreasing with increasing temperature. Together with the NORSOK ranking of the material (Fig. 1(c)) an interesting material behavior was observed, a decreasing volume change with an increasing NORSOK ranking for a rising testing temperature.



Fig. 1. Volume increase during (a) compression and (b) during decompression for HNBR1 depending on the testing temperature at a saturation pressure of 150bar, pure CO₂ and a depressurization rate of 100bar/min. (c) NORSOK material ranking for cylindrical specimens tested at several temperatures (Numbers in the right upper corner represents different measurements).

To explain this material behavior the model of free volume according to G. W. Ehrenstein (2011) for polymers should be taken into considerations. With rising temperature the polymer chains easily align in loading direction with rising temperatures leading to an earlier break of the material. This assumption may explain the decreasing volume change with increasing NORSOK ranking due to the earlier crack initiation and breakdown of the material at higher temperature.

3.2. Influence of the testing gas media

For the investigation of the influence of different gases on the material performance, experiments were implemented with CO_2 and CH_4 as well as mixtures of both. Figure 2(a) shows the volume increase during the compression phase depending on the CO_2 content for HNBR1 at a temperature of 90°C and a saturation pressure of 100bar. The volume change clearly increases with rising CO_2 content. This increase can be explained with the two effects occurring during exposure of materials to high pressure gas, the plasticization of the matrix due to the gas and the compression of the matrix because of the ambient pressurization. Carbon dioxide acts as a strong plasticizer whereas the compression effect is more considerably for methane.



Fig. 2. Volume increase during (a) compression and (b) during decompression for HNBR1 depending on the carbon dioxide content at a saturation pressure of 100bar, a temperature of 90°C and a depressurization rate of 100bar/min. (c) NORSOK material ranking for cylindrical specimens and components tested with several gas mixtures (Numbers in the right upper corner represents different measurements).

The volume change during the decompression phase depending on the CO_2 content is summarized in Fig. 2(b) for a temperature of 90°C, a saturation pressure of 100bar and a depressurization rate of 100bar/min. With increasing CO_2 content a clear increasing volume change was monitored. This can be explained by the amount of solved gas in the material. The testing conditions drives CO_2 to supercritical conditions, as described by F. Rindfleisch et al. (1996) and therefore, the highest amount of gas is solved for pure CO_2 leading to the highest volume increase. This assumption is also intensified with an increasing NORSOK ranking (Fig. 2(c)) for both, cylindrical specimens and tested components (O-ring). Based on the high amount of gas in the material and the spontaneous interruption of the established equilibrium after the saturation phase the gas cannot diffuse out of the material. Therefore, a higher volume change is recorded with more amount of solved gas leading to a worse NORSOK ranking.

3.3. Influence of saturation pressure

For the investigation of the saturation pressure on the material performance experiments were carried out at several pressures (50bar, 100bar and 150bar). Figure 3(a) shows the volume increase during the compression phase depending on the saturation pressure at a temperature of 90°C for pure CO₂. Based on Henry's law, W. Henry (1802), the gas concentration in the material rises with increasing saturation pressure and therefore more gas is solved in the material combined with a stronger plasticization effect leading to a higher volume change during compression for higher saturation pressures. The relative volume change during the depressurization depending on the saturation pressure is depicted in Fig. 3(b) for a temperature of 90°C, pure CO₂ and a decompression rate of 100bar/min.



Fig. 3. Volume increase during (a) compression and (b) during decompression for HNBR1 depending on the saturation pressure at a temperature of 90°C, pure CO₂ and a depressurization rate of 100bar/min. (c) NORSOK material ranking for cylindrical specimens and components tested at several saturation pressures (Numbers in the right upper corner represents different measurements).

For a saturation pressure of 50bar nearly no volume change compared to the other pressure states was recorded. As mentioned above the amount of solved gas strongly depends on the applied pressure and therefore a higher amount of gas is solved for higher pressure leading to higher volume change and finally a worse NORSOK ranking for both, cylindrical specimens and component tests (Fig. 3(c)).

3.4. Influence of depressurization rate

Concerning different testing standards, various decompression rates were recommended. To investigate the influence of different decompression rates on the material performance experiments with a depressurization rate of 20, 40, 60, 80 and 100bar/min were implemented. Based on the same saturation conditions for all experiments the same amount of gas is dissolved for the depressurization study. Figure 4(a) shows the relative volume change during the depressurization process depending on the decompression rate for pure CO₂, a temperature of 90°C and a saturation pressure of 150bar. As depicted a clear increase of the observed volume change with increasing depressurization rate

was monitored. This effect can be explained with the lack of time for the establishment of a permeation process for higher depressurization rates. Additional to the maximum volume change, the change in the kinetics was investigated as well. Figure 4(b) summarizes the initial time difference, needed for starting the volume change depending on the used depressurization rate. A strong influence on the initial time difference was recorded in the region of 20 to 60bar/min, whereas at higher depressurization rates, the influence seems only marginal.



Fig. 4. Volume increase during (a) decompression and (b) initial time difference to start a volume change during decompression for HNBR1 depending on the depressurization rate at a temperature of 90°C, pure CO₂ and a saturation pressure of 150bar. (c) NORSOK material ranking for cylindrical specimens tested with several depressurization rates (Numbers in the right upper corner represents different measurements).

In contrast to the clear influence of the decompression rate on the volume change and the kinetics of the volume change, the NORSOK ranking seems similar for all investigated depressurization rates (Fig. 4(c)). Due to the observed differences within the reproducibility measurements, the volume change during the depressurization should be considered critically by comparing different testing parameters and different materials.

3.5. Considerations to the observed volume change during depressurization

The observed volume change during the depressurization step seems useful for the improvement of the material knowhow and a material related design process. Nevertheless, the resulting volume change is the result of a highly complex process and a result of several processes happening simultaneous during the exposure to high temperature, high pressure and several gases. Therefore, a direct correlation between the volume change during the experiment and the empirical established NORSOK ranking could not be established.

4. Conclusions

Within the experimental investigations, a autoclave testing device equipped with a camera system was used to measure the impact of several influencing parameters on the volume change during the compression and the decompression phase. The observed volume change was correlated with the material ranking concerning NORSOK testing standard (2001). The increase of the testing temperature leads to a decreasing volume change during the depressurization process combined with a rising NORSOK ranking of the material. This behavior could be explained with the earlier crack initiation and breakdown of the material at higher temperature. Beside the temperature, also the used gas has a strong influence on the material performance. With increasing carbon dioxide content a strong pronounced rise of the saturation volume as well as increasing volume change during the depressurization was recorded. The observed NORSOK ranking rises as well, explainable with the higher amount of solved gas in the material. Similar to the rising volume change with rising CO_2 content, the volume change and the NORSOK ranking rises for higher saturation pressures. This behavior can be explained with the pressure dependency of the solubility following Henry's law, W. Henry (1802). Finally, the influence of different depressurization rates was investigated.

Whereas, a strong influence on the volume change and the kinetics of the volume change during decompression was observed, the NORSOK ranking changes only marginal. Therefore, a simple connection between the volume change and the observed rapid gas decompression performance could not be established due to the observed tendencies. Although, the measurements lead to a better knowledge of the material behavior and support a material related design process.

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